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PATENT

41928

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Yukihiro MATSUMOTO et al.

Serial No.: 09/883,266

Filed: June 19, 2001

For: **PRODUCTION PROCESS FOR
WATER-ABSORBENT RESIN**

Attention: Box Missing Parts

RESPONSE TO NOTICE OF INCOMPLETE REPLY

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

This is in reply to the Notice of November 21, 2001.

The November 21, 2001 Notice incorrectly states that English translations of the "drawings" were not filed with Applicant's September 27, 2001 response. The original specification included a single drawing (Figure 1) without any written text.

Appended to the original Japanese specification were Tables 1-3. The Tables 1-3 are incorporated into the verified English translation and the substitute specification as filed on September 27, 2001. The English translation of Table 1 appears on page 34 of the verified English translation and page 33 of the substitute specification. The English translation of Table 2 appears on page 38 of the verified translation and page 37 of the substitute specification. The English translation of Table 3 appears on page 44 of the verified translation and page 43 of the substitute specification.

In view of the above, Applicants' September 27, 2001 Response to the Notice of Missing Parts was complete. Enclosed are copies of pages 34, 38 and 44 of the verified translation, and pages 34, 37 and 43 of the substitute specification as filed on September 27, 2001.

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Regarding the fee under 37 C.F.R. § 1.17(k), Applicants' response expressly authorized any fee deficiencies under 37 C.F.R. § 1.116 and 37 C.F.R. § 1.17 be charged to Deposit Account 18-2220. Thus, to the extent there are any fee deficiencies, they should be charged to the Deposit Account.

In view of the above, Applicants' September 27, 2001 response was complete. In the event other fees are due, the Commissioner is hereby authorized to charge any fees in connection with this reply, including extension fees and processing fees under 37 C.F.R. § 1.16 and 37 C.F.R. § 1.17 to our Deposit Account No. 18-2220.

Respectfully submitted,



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47 minutes. The resultant crosslinked hydrogel polymer (6) was cut into pieces of the size of about 1 mm, and then mixed with sodium carbonate powder so as to have a neutralization ratio of 75 mol %, and then dried, pulverized, and classified in the same way as of Example 3, thus obtaining a water-absorbent resin powder (6).

[Table 1]

	Polymerization container	Protoanemonin content (ppm)	Polymerization peak		Water absorption capacity (g/g) without load	Water-extractable content (weight %)	Residual monomer (ppm)
			Time (minutes)	Temperature (°C)			
Example 1	Kneader	1	23	78	33	5	280
Comparative Example 1	Kneader	30	27	80	33	7	470
Example 2	Vat	3	8	96	34	6	120
Comparative Example 2	Vat	13	12	102	34	8	160
Example 3	Cylindrical container	1	16	98	51	22	160
Example 4	Cylindrical container	3	18	103	53	25	480
Example 5	Cylindrical container	9	24	98	55	26	620
Comparative Example 3	Cylindrical container	17	29	93	56	28	760
Comparative Example 4	Cylindrical container	17	Weak-alkali treatment / no polymerization				
Comparative Example 5	Cylindrical container	(Unneutralized) 17	No alkali treatment / no polymerization				
Example 6	Cylindrical container	(Unneutralized) 3	47	104	54	10	980

As is understood from comparisons of Examples 3 to 5 with Comparative Examples 3 to 5 under the same polymerization conditions in Table 1, as the protoanemonin content increases from 1 ppm to 17 ppm, the polymerization time (time as needed till the peak) gets longer and the residual monomer content increases, and further, the water-extractable content greatly increases when compared with a small increase of the water absorption capacity, so the properties are relatively deteriorated. In addition, as is also understood from comparisons among Comparative Examples 3 to 5 and comparisons between Examples 2 and 6, it is understood that even if the same acrylic acid is used, the alkali treatment, particularly, strong-alkali treatment, greatly enhances the polymerizability.

Also shown in Table 1 are the times which passed until the polymerization peak temperatures were indicated in the polymerization processes to obtain the

according to the present invention is greatly decreased.

[Table 2]

	Surface-crosslinked water-absorbent resin powder	Water absorption capacity (g/g) without load	Water absorption capacity (g/g) under load (4.90 kPa/1.96 kPa)	Liquid permeation quantity (g) under load
Example 7	Water-absorbent resin powder (7)	26	24/26	525
Comparative Example 6	Comparative water-absorbent resin (6)	26	24/26	465
Example 8	Water-absorbent resin powder (8)	26	24/26	—
Comparative Example 7	Comparative water-absorbent resin powder (7)	26	23/25	—
Example 9	Water-absorbent resin powder (9)	42	25/38	—
Comparative Example 8	Comparative water-absorbent resin powder (8)	40	22/35	—
Example 10	Water-absorbent resin powder (10)	43	25/38	—

The measurement methods for the data of Table 2 are as follows:

Water absorption capacity under load:

The water absorption capacity of the water-absorbent resin powder for physiological saline solution under a load of 50 g/cm² (about 4.90 kPa) or 20 g/cm² (about 1.96 kPa) was measured in accordance with the methods as disclosed in the working examples portions of EP 0885917, EP 0811636, and USP 6207772B, namely, in the following way.

An amount of 0.900 g of water-absorbent resin powder was allowed to absorb a physiological saline solution over a period of 60 minutes in a state where a load of 50 g/cm² (about 4.90 kPa) or 20 g/cm² (about 1.96 kPa) was uniformly applied to the water-absorbent resin powder, and then the weight W2 (g) of the absorbed physiological saline solution was measured with a balance. From this weight W2, the water absorption capacity (g/g) under the load was calculated in accordance with the following equation:

water absorption capacity (g/g) under load

$$= (W2 \text{ (g)}/\text{weight (g) of water-absorbent resin powder}).$$

glycol/water = 0.1/3/1 (weight % based on water-absorbent resin powder) was added by spraying to 500 g of the water-absorbent resin powder (14) as obtained in Example 14, and the resultant mixture was heated under stirred conditions in an oil bath for 30 minutes, thus obtaining a water-absorbent resin powder (16). The results are shown in Table 2.

[Table 3]

	Strong-alkali treatment time (minutes)	Polymerization peak/temperature (°C)	Water absorption capacity (g/g) without load	Water-extractable content (weight %)	Color of resin
Example 10	100 mol % * 20-30°C	145/83	48	12	Yellow
Example 11	105 mol % * 20-30°C	140/70	44	12	Yellow
Example 12	105 mol % * 20-30°C + rt. 16hrs	75/87	52	18	Yellow
Example 13	105 mol % * 40-50°C	40/95	55	23	Light yellow
Example 14	105 mol % * 40-50°C + rt. 16hrs	37/99	65	23	Light yellow
Example 15	105 mol % * 40-50°C + 50°C * 16hrs	22/104	57	24	Light yellow
Comparative Example 9	None	(It took not less than 5 minutes to initiate the polymerization.)			
Comparative Example 10	Weak-alkali treatment	(It took not less than 5 minutes to initiate the polymerization.)			

As is understood from Table 3, in the case where the acrylic acid having a furfural content of 230 ppm was not subjected to alkali treatment, the progress of the polymerization is so slow as not to be fit for the production (Comparative Examples 9 & 10).

On the other hand, in Examples 10 to 15 in which strong-alkali treatments were carried out, the polymerization runs in about 1 minute (it took 145 minutes to attain the peak in Example 10). Furthermore, if the temperature or neutralization ratio in the strong-alkali treatment is raised to prolong the time, then the polymerization time is greatly shortened (to 37 minutes in Example 14/to 22 minutes in Example 15), and the residual monomer content is also reduced, and, in Example 15, the polymerizability is exhibited to almost the same degree as acrylic acid having an aldehyde content of less than 10 ppm. Furthermore, as it goes on from Examples 10 to 15 in which the alkali treatment was more and more strengthened in this order, the

Table 2

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Example 7	Water-absorbent resin powder (7)	26	24/26	325
Comparative Example 6	Comparative water-absorbent resin (6)	26	24/26	465
Example 8	Water-absorbent resin powder (8)	26	24/26	—
Comparative Example 7	Comparative water-absorbent resin powder (7)	26	23/25	—
Example 9	Water-absorbent resin powder (9)	42	25/38	—
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An amount of 0.900 g of water-absorbent resin powder was allowed to absorb a physiological saline solution over a period of 60 minutes in a state where a load of 50 g/cm² (about 4.90 kPa) or 20 g/cm² (about 1.96 kPa) was uniformly applied to the water-absorbent resin powder, and then the weight W2 (g) of the absorbed physiological saline solution was measured with a balance. From this weight W2, the water absorption capacity (g/g) under the load was calculated in accordance with the following equation:

water absorption capacity (g/g) under load

$$= (W2 \text{ (g)}/\text{weight (g) of water-absorbent resin powder}).$$

Liquid permeation quantity under load:

With a measurement apparatus of Fig. 1 used, 0.900 g of water-absorbent resin

Example 14, and the resultant mixture was heated under stirred conditions in an oil bath for 30 minutes, thus obtaining a water-absorbent resin powder (16). The results are shown in Table 2.

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	Strong-alkali treatment time (minutes)	Polymerization peak time (minutes)/temperature (°C)	Water absorption capacity (g/g) without load	Water-extractable content (weight %)	Color of resin
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Example 11	105 mol % * 20-30°C	140/70	44	12	Yellow
Example 12	105 mol % * 20-30°C + rt. 16hrs	75/87	52	18	Yellow
Example 13	105 mol % * 40-50°C	40/95	55	23	Light yellow
Example 14	105 mol % * 40-50°C + rt. 16hrs	37/99	65	23	Light yellow
Example 15	105 mol % * 40-50°C + 50°C * 16hrs	22/104	57	24	Light yellow
Comparative Example 9	None	(It took not less than 5 minutes to initiate the polymerization.)			
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As is understood from Table 3, in the case where the acrylic acid having a furfural content of 230 ppm was not subjected to alkali treatment, the progress of the polymerization is so slow as not to be fit for the production (Comparative Examples 9 & 10).

On the other hand, in Examples 10 to 15 in which strong-alkali treatments were carried out, the polymerization runs in about 1 minute (it took 145 minutes to attain the peak in Example 10). Furthermore, if the temperature or neutralization ratio in the strong-alkali treatment is raised to prolong the time, then the polymerization time is greatly shortened (to 37 minutes in Example 14/to 22 minutes in Example 15), and the residual monomer content is also reduced, and, in Example 15, the polymerizability is exhibited to almost the same degree as acrylic acid having an aldehyde content of less than 10 ppm. Furthermore, as it goes on from Examples 10 to 15 in which the alkali treatment was more and more strengthened in this order, the water absorption capacity is also more and more enhanced, and coloring of the

VERIFICATION OF TRANSLATION

I, Takehiko MATSUMOTO, c/o Matsumoto & Associates of 25-6, Hannan-cho 1-chome, Abeno-ku, OSAKA 545-0021 JAPAN, am proficient in both the English and Japanese languages and I personally prepared the English-language translation of the attached document, I certify that to the best of my knowledge and belief the translation is a complete and accurate translation of the Japanese document into the English language.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

September 17, 2001

Date



Takehiko MATSUMOTO